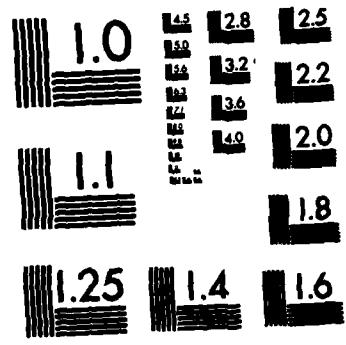


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Chemical Deposition of TiO₂ Layers
on GaAs

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This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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chemical structure of the films are determined by the amount of water present in the coating solution, coating atmosphere, and hydrolysis atmosphere. Films grown in high humidity exhibit much intermixing of semiconductor and oxide components, and oxidation of the semiconductor; films grown in low humidity exhibit neither mixing nor oxidation of GaAs unless they are stored in that atmosphere for a prolonged period.

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I. INTRODUCTION

GaAs is an important semiconductor material that can be employed in high-speed electronic devices such as microwave oscillators and high-frequency field-effect transistors (FETs). Experimental high-speed GaAs devices intended for incorporation into microwave systems--devices such as counters, analog-to-digital converters, multipliers, and memories--have been constructed with FETs based on metal-semiconductor contacts and Schottky diode technologies. When compared with a projected technology based on metal-insulator-semiconductor (MIS) devices, however, these two technologies are seen to have disadvantages. Schottky diode devices have a higher power dissipation than MIS devices. Metal-semiconductor-based FETs have low (<0.8 V) logic swings, whereas an MIS device would have a 1 V swing, which improves accuracy in digital circuits.

Production of stable GaAs metal-oxide-semiconductor (MOS) devices has been hindered by the poor quality of the native oxide. Use of nonnative oxides or other passivants to form the MIS system could remove this hindrance. Many nonnative insulating layers have been produced on compound semiconductors: SiO_2 layers on InP by chemical vapor deposition (CVD) and plasma-enhanced CVD;¹ SiO_2 on InSb by low-temperature CVD;² Al_2O_3 on InP by pyrolysis;³ Si_3N_4 on GaAs by plasma-enhanced CVD;⁴ and AsOF on GaAs by glow-discharge plasma.⁵ But electronic instabilities have been detected in these insulators as well. The presence of a thin layer of native oxide between the insulator and the semiconductor has been indicated as the cause of the electronic instabilities.¹⁻⁴

Room-temperature chemical solution deposition of TiO_2 was selected as a demonstration system to explore the possibility of placing a nonnative oxide layer on GaAs without an interfacial layer of native oxide and without intermixing of semiconductor and oxide components. Room temperature reduces the possibility of diffusion and mixing at the semiconductor-oxide interface. There are two reasons for selecting chemical solution deposition. First, previous work⁶ provides procedures on how to control the initial GaAs surface in a solution process and, in particular, how to remove the native oxide and transfer the cleaned sample to another solution without reoxidation. Second, the particular deposition reaction chosen, hydrolysis of

Ti alkoxides, has been widely studied on oxide substrates⁷ and is in fact a commercial process (using, for example, Dupont TYZOR organic titanates). Initially it was not apparent that a nonnative oxide would adhere to the semiconductor unless an intervening layer of native oxide was present. For this reason, the well-characterized reaction of Ti alkoxides to form TiO_2 was preferred to the almost uncharacterized reaction of Si alkoxides to form SiO_2 ,⁷ even though formation of a device-quality insulating layer was not expected. The deposition technique consists of dipping the substrate into a solution of an organometallic compound [e.g., M(OR)_4 where M = Ti in this case and R = isopropyl or 2-ethylhexyl], then hydrolyzing the organometallic layer on the substrate to produce the metal oxide.

The experimental conditions in both the coating and the hydrolysis steps affect the chemical composition of films produced by the deposition technique.⁶ In particular, the presence of water in the hydrolysis atmosphere determines the amount of oxidized Ga and As in the TiO_2 insulating film. The amount of such admixture may be crucial to the electrical characteristics of the film: a highly mixed oxide layer may have many undesirable charge traps.

X-ray photoelectron spectroscopy (XPS) was used to characterize the chemical compositions of the metal oxide films that had been exposed to different humidities for various lengths of time. Depth profiles of the films were obtained either by sputtering the surface with energetic Ar^+ ions or by a chemical-etch procedure.

II. EXPERIMENTAL

The GaAs coupons are polished (100) faces of p-type material, Zn-doped to a carrier concentration of $5 \times 10^{18} \text{ cm}^{-3}$. They were etched in 2% Br₂ in methanol for 15 sec, rinsed in methanol, then immersed in a 3.5 vol% solution of titanium isopropoxide or titanium 2-ethylhexoxide in isopropanol. All samples were kept in methanol and not allowed to dry as they were introduced into the titanate solution, a procedure reported to prevent oxidation.⁶ The samples were immersed in the coating solution for 1 min, then removed and hydrolyzed while lying horizontally on microscope slides. Relative humidity (RH) of 95% was obtained in stagnant air over a water bath (lower humidity was characteristic of stagnant room air). Samples 12 and 13 were placed in a desiccator over CaSO₄ immediately after they were removed from the coating solution. A summary of the samples is given in Table I.

The samples remained in their hydrolysis atmospheres until analysis, which generally occurred within one week, except as noted in Table I. The x-ray photoelectron spectra were obtained with a GCA-McPherson ESCA-36 spectrometer equipped with an electrostatic spherical sector analyzer and a position-sensitive detector. The sensitivity of the spectrometer is approximately 10% of a monolayer at the surface. Auger spectra were obtained with a Phi 590 scanning Auger microprobe.

Energetic Ar⁺ ion sputtering was used at first to determine the thickness and depth profiles of the films. The slow sputter rate of the XPS Ar gun ($\sim 5 \text{ \AA/min}$) resulted in unacceptably long times for a complete depth profile, since the films are 500 to 2000 Å thick. For this reason, a chemical-etching procedure developed at The Aerospace Corporation for etch profiling Si/SiO₂ structures was extended for use on the current samples. The etching solution is 2 parts HF : 25 parts NH₄F : 5 parts H₂O. The sample is immersed in the solution for 1 sec, removed, rinsed with methanol, and loaded into the spectrometer. The entire procedure takes place in a glove bag attached to the spectrometer. The spectrometer and the glove bag are filled with flowing N₂ to eliminate oxidation of the etched surface until the spectrometer is evacuated.

The solution-etching method has several advantages over ion sputtering. First, etch rate is approximately 50 Å/sec, so the experiments can be

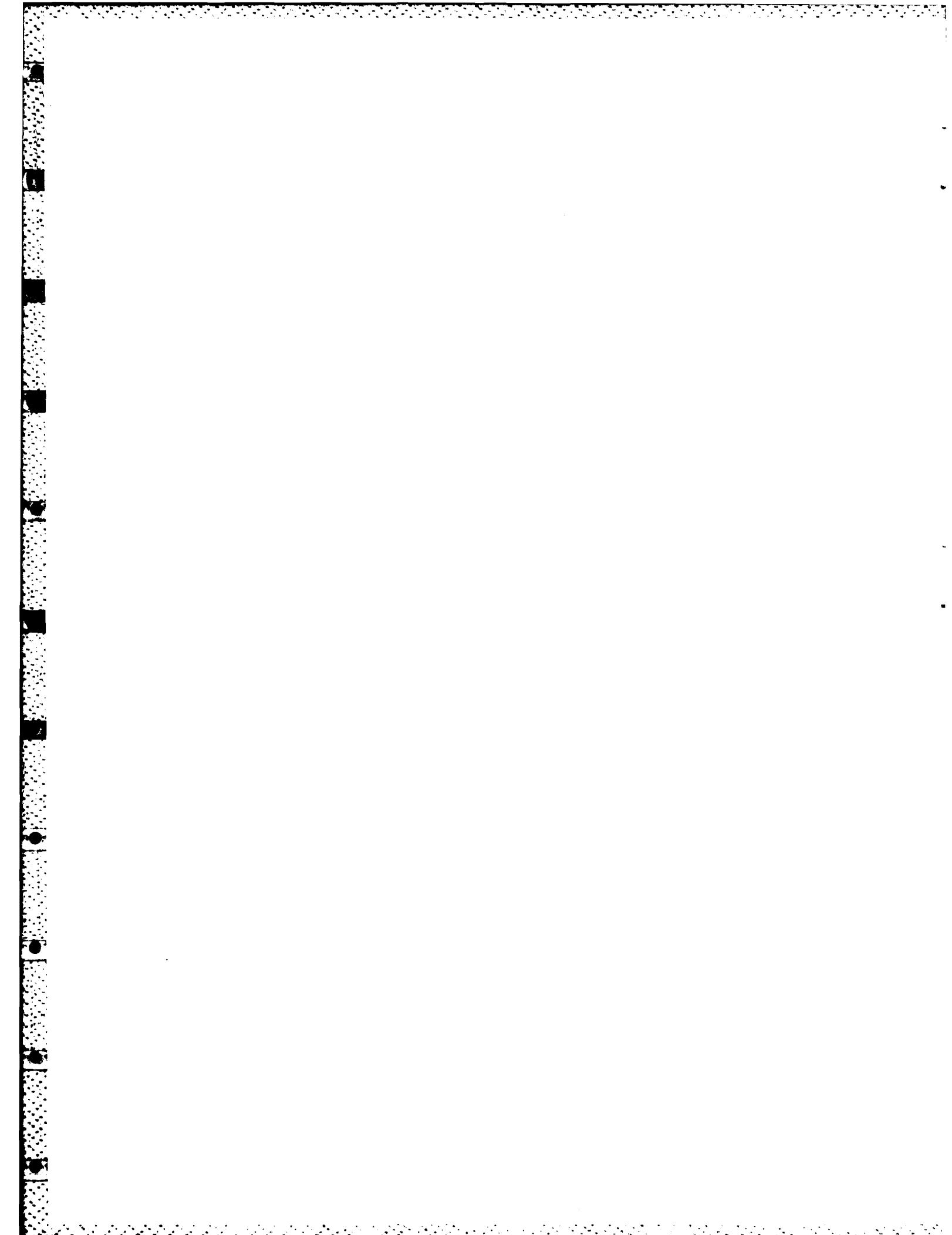
TABLE I. DESCRIPTION OF SAMPLES

Sample	Alkoxide	Hydrolysis conditions	Results
1	isopropyl	20% RH	No mixing
2	isopropyl	95% RH	Mixed oxide
3	isopropyl	25% RH	No mixing
4	isopropyl	25% RH	No mixing
5	2-ethylhexyl	95% RH	Mixed oxide
6	2-ethylhexyl	30% RH, over 1 month ^a	Some mixing
7	2-ethylhexyl	30% RH, 1 month ^a	No mixing
8	2-ethylhexyl	30% RH, 3 days ^a	No mixing
9	2-ethylhexyl	95% RH	Mixed oxide
10	2-ethylhexyl	30% RH, 1 week ^a	No mixing
11	2-ethylhexyl	30% RH, 2 weeks ^a	No mixing
12	2-ethylhexyl	Desiccator	No mixing
13	2-ethylhexyl	Desiccator	No mixing

^aLength of time in hydrolysis atmosphere before analysis.

completed in a reasonable length of time. Second, and more important, the damage associated with sputtering by high-energy (10 keV) Ar⁺ ions has been avoided. Some well-known forms of ion-beam damage are preferential sputtering, reduction of surface compounds (reduction of TiO₂ was observed in these experiments), deposition of C contamination, crater formation, and ion knock-on and other effects that broaden the measured film-substrate interface. These effects make it impossible to measure accurately a small amount of an interfacial compound after several surface layers have been removed: the interface region is too mixed. In solution etching, these mixing effects do not occur since the etch is a less energetic process than ion-beam sputtering.

The etchant for the solution-etch procedure must be carefully selected so that preferential etching by the solution or possible reactions that could leave new compounds on the surface do not occur. In some cases, F was found on the surface of the samples after etching, but was associated with Ti and not Ga or As, as evidenced by a binding-energy shift for Ti when F was present. Proper rinsing eliminated that problem. Etch-pit formation must be minimized, since it causes the interface to appear broad and mixed even if it is actually abrupt, and decreases the amount of interfacial material that can be detected. Microscopic observation of etched samples reveals the presence of etch pits and leads to an estimate of a 10-fold reduction in the area of the film with constant thickness, and thus in the signal of the interfacial material, which is proportional to that area. A faster etch will produce fewer pits, but the depth resolution will also decrease. The present system is a good compromise between interface broadening due to etch-pit formation and etch rate.



III. RESULTS AND DISCUSSION

XPS reveals elemental compositions and, by means of photoelectron binding energies, information about the chemical state of each element in the sample. If the sample is an insulator, or is not in good electrical contact with the spectrometer, electrostatic charging can cause apparent binding-energy shifts. Referencing all binding energies to a single element present on the sample, such as adventitious C, usually compensates for these shifts. But because that convenient reference material is removed by sputtering and is often severely reduced in intensity by chemical etching, binding-energy differences rather than absolute binding energies are used in this study. Standard binding-energy differences and representative binding-energy differences measured in these experiments are listed in Tables II and III, respectively. Samples 1 and 3 were hydrolyzed in low-humidity environments and, when the peaks appear after removal of the TiO_2 layer, have a Ga(3d)-As(3d) binding-energy difference characteristic of GaAs (21.9 eV). Sample 2, hydrolyzed in 95% relative humidity, shows Ga and As with a binding-energy difference (25.0 eV) indicative of Ga_2O_3 and As_2O_3 in the bulk of the TiO_2 film. After 200 Å of the film is removed, the substrate GaAs is visible (binding-energy difference of 21.9 eV).

Depth profiles and XPS spectra for typical films produced with 2-ethylhexyl titanate and hydrolyzed in relative humidities of 95% and 30% are shown in Figures 1 and 2, respectively. The ordinate, XPS peak intensity, is proportional to the amount of a given species present. Peak-intensity values have been corrected for spectrometer sensitivity, but not for electron escape depth (which alters the volume sampled for each element) or for preferential etching. The peak-intensity scale, therefore, is given in arbitrary units, and the relative magnitudes of the plotted peak intensities for different elements should not be compared. The trends in intensity with etching time are the salient features of these depth profile plots.

The plots and Table III reveal readily apparent differences in the effects of the two hydrolysis atmospheres: the high-humidity film is a mixed oxide throughout its bulk, whereas the low-humidity film is a titanium oxide only. Single oxide layers were found for samples that had been stored in a desiccator containing $CaSO_4$ from immediately after preparation until analysis,

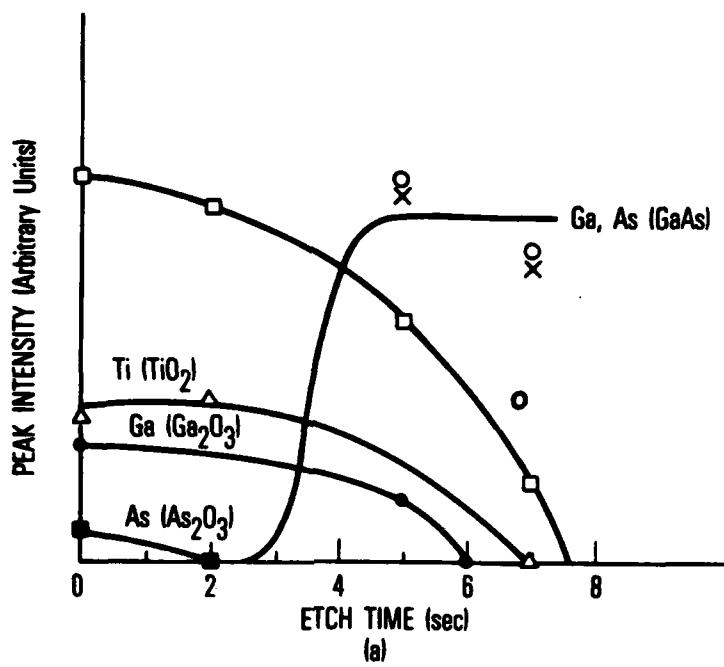
TABLE II. STANDARD BINDING-ENERGY DIFFERENCES

Sample	Ga(3d)-As(3d) (eV)	Ga(1)-Ga(2)	As(1)-As(2) (eV)	References
GaAs	21.9			a
	22.0			b
	21.8			c
GaAs-Ga ₂ O ₃	20.7	1.2		a
	20.9	0.9		c
As-As ₂ O ₃			3.8	a
			3.7	b
			3.2	c
Ga ₂ O ₃ -As	21.3			a
	21.1			c
Ga ₂ O ₃ -As ₂ O ₃	25.1			a
	24.3			c
Ga ₂ O ₃ -As ₂ O ₅	26.1			a
	25.8			c
GaAs-As	22.4		0.3	a
	22.5		0.5	b
	22.4		0.6	c
GaAs-As ₂ O ₃	26.2		4.3	a
	26.2		4.2	b
	25.6		3.8	c
GaAs-As ₂ O ₅	27.2		5.3	a
	27.1		5.3	c

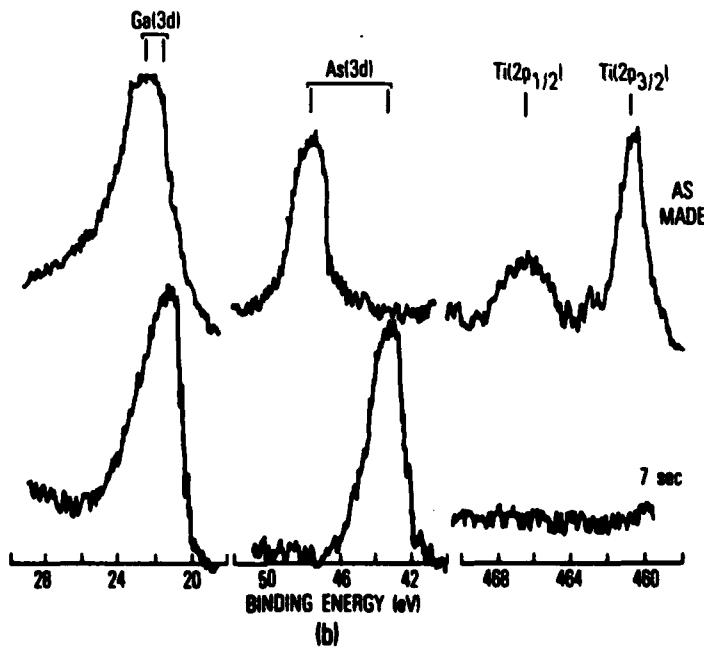
^aP. A. Bertrand, *J. Vac. Sci. Technol.* **18**, 28 (1981).

^bY. Mizokawa, H. Iwasaki, R. Nishitani, and S. Nakamura, *J. Electron Spectrosc. Relat. Phenom.* **14**, 129 (1978).

^cC. D. Wagner, W. M. Riggs, L. E. Davis, and J. F. Moulder, *Handbook of X-ray Photoelectron Spectroscopy*, Perkin-Elmer Corporation, Eden Prairie, Minnesota (1979).



(a)



(b)

Fig. 1. Titanium oxide film on GaAs prepared with hydrolysis at 95% RH (sample 9): (a) depth profile, chemical etch; (b) typical XPS spectra taken during depth profiling.

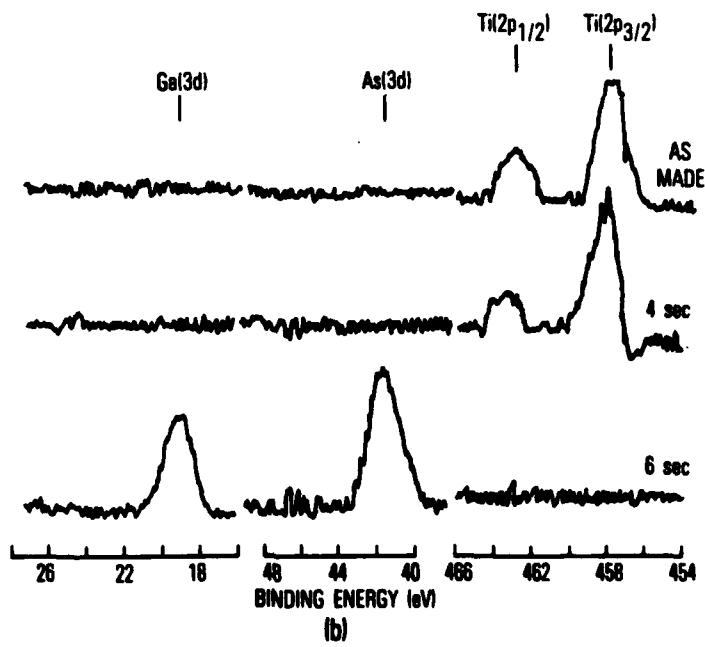
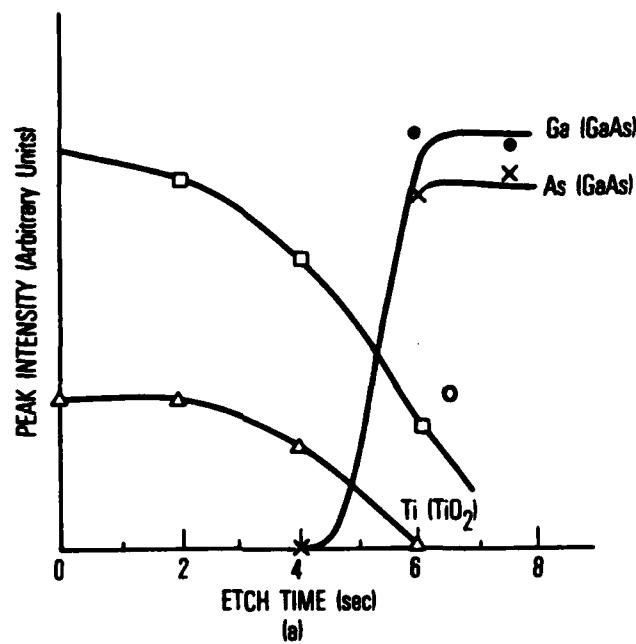


Fig. 2. Titanium oxide film on GaAs prepared with hydrolysis at 30% RH for two weeks (sample 11): (a) depth profile, chemical etch; (b) typical XPS spectra taken during depth profiling.

and for samples that had been hydrolyzed in room air for two weeks or less before analysis. The high-humidity samples contained Ga_2O_3 and As_2O_3 in their oxide layer.

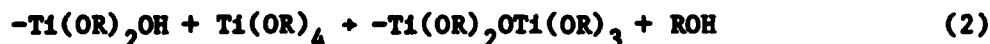
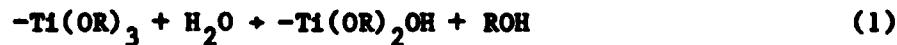
TABLE III. EXPERIMENTAL BINDING-ENERGY DIFFERENCES

Sample	Ga(3d)-As(3d) (eV)
1	21.9
2	25.0
2 ^a	21.9
3	21.8

^aBelow 200 Å.

The compositions of the films produced with titanium isopropoxide depend on humidity of the hydrolysis atmosphere in the same manner as do those produced with titanium 2-ethylhexoxide. The depth profile of sample 4 (a low-humidity hydrolysis film) in Figure 3 exhibits no mixing of Ga or As into the TiO_2 film. The film is approximately 2000 Å thick, whereas the titanium 2-ethylhexoxide films are only ~800 Å thick, a difference explained by the mechanism of film formation.

Water is required for the hydrolysis of titanium alkoxides to TiO_2 , which proceeds as follows:⁷



where R is the alkoxide group, and the dangling bond on Ti represents connection to the surface. These two reactions are repeated to build up the oxide coating. Water for the reactions is present as vapor in the hydrolysis and coating atmospheres and is dissolved in the coating solution [most of

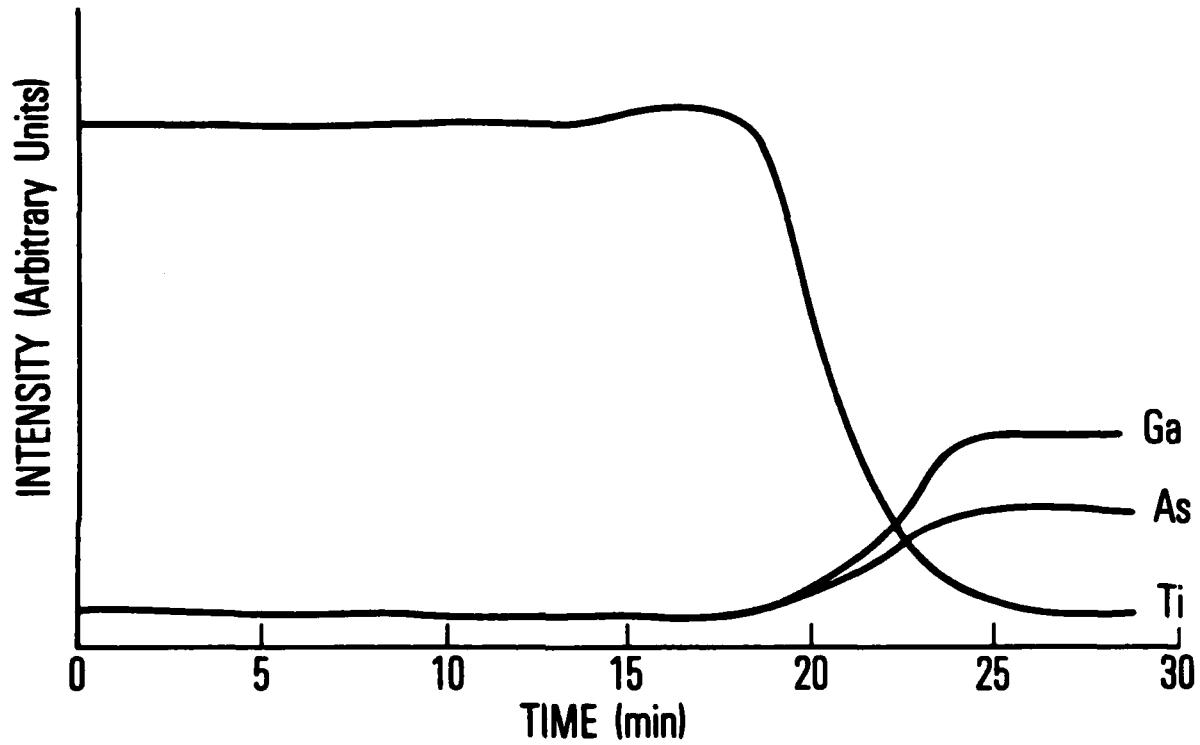


Fig. 3. Titanium oxide film on GaAs prepared with hydrolysis at 25% RH (sample 4). Depth profile by scanning Auger microprobe with sputter etching.

which is scavenged by $Ti(OR)_4$ in the solution]. Two forms of alkoxide are represented in the above equations: (1) alkoxide strongly bound to the substrate surface and (2) alkoxide loosely associated with the surface in an unconsolidated layer. The samples are removed vertically from the solution, which permits some of the unconsolidated layer to drain off before the samples are positioned horizontally for the remainder of the hydrolysis time. The isopropoxide is much more reactive in hydrolysis than the 2-ethylhexoxide, and probably forms a thicker layer of bound alkoxide during coating. This thick bound-alkoxide layer will then form a thick TiO_2 layer during hydrolysis.

It is well known that the presence of H_2O enhances the oxidation of GaAs to Ga_2O_3 , As_2O_3 , and As .⁸ High-humidity hydrolysis of Ti alkoxides yields thick, mixed-oxide coatings on Al surfaces, owing to prepolymerization of the alkoxide and the interaction of water with the Al surface.¹ In high humidity, oxidation of GaAs and hydrolysis of the Ti alkoxide occur simultaneously, producing a network of mixed Ga, As, and Ti oxides. If the amount of water present is insufficient to form the mixed oxide, the hydrolysis reaction occurs preferentially. The thermodynamic stabilities of Ga_2O_3 and TiO_2 are similar with respect to their elements, so the preferential hydrolysis must be due to kinetic effects: the hydrolysis reaction must be faster than oxidation at room temperature in low humidity.

A low-humidity environment does not totally preclude the formation of Ga and As oxides, however, as Figure 4 demonstrates for sample 6, which was hydrolyzed and stored in room air for over one month before analysis. The presence of Ga_2O_3 and As_2O_3 near the surface evidences that even a small amount of water vapor in the storage atmosphere can cause mixed oxides to form during a prolonged storage period. Thus, with long exposure to even relatively low humidity, the substrate GaAs begins to oxidize. Oxidized semiconductor components are found at the surface of the oxide, indicating that the free energy of the oxide system for these low concentrations of oxidized Ga and As is lower with a Ga_2O_3 - As_2O_3 layer at the surface than with a uniform Ga and As oxide distribution throughout the oxide or with a Ga_2O_3 - As_2O_3 layer at the oxide-semiconductor interface. Whether the diffusion of Ga and As proceeds before or after the oxidation cannot be determined from these

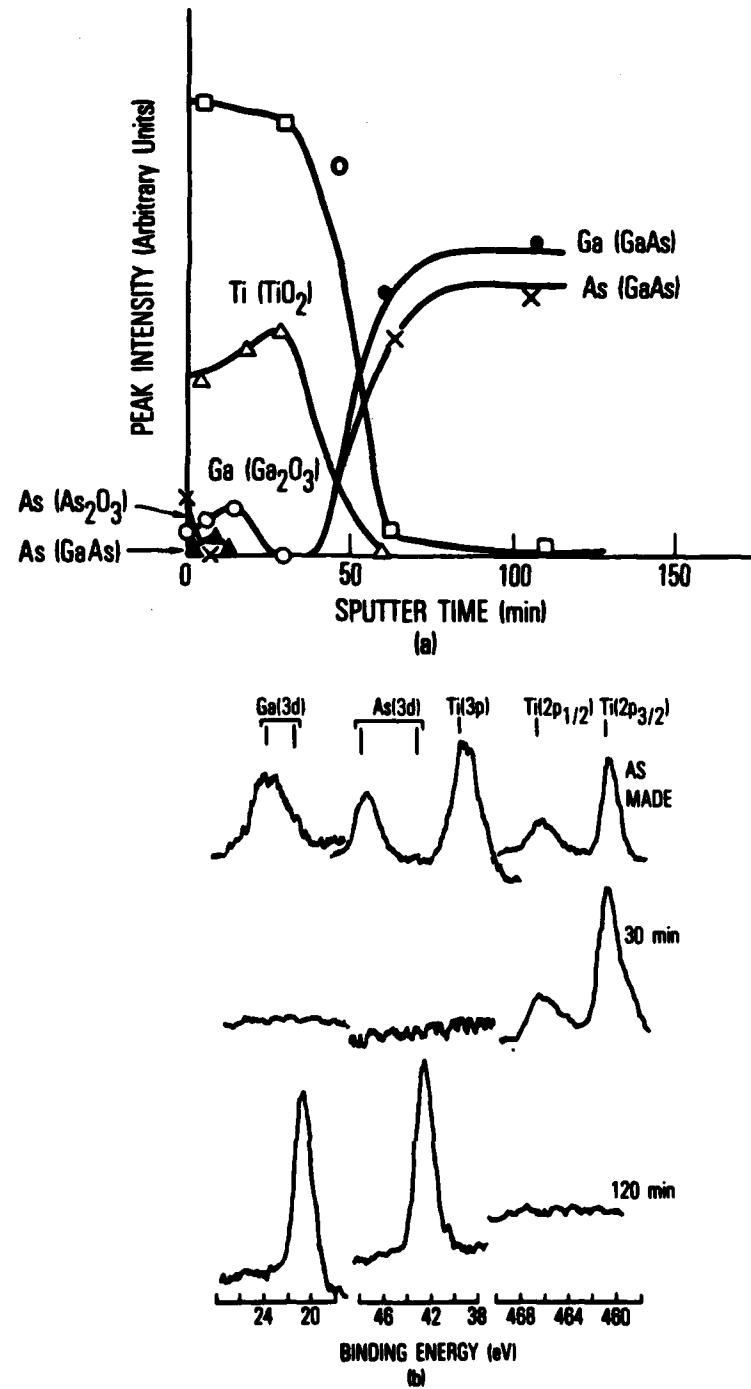


Fig. 4. Titanium oxide film on GaAs prepared with hydrolysis at 30% RH for over one month (sample 6): (a) depth profile, sputter etch; (b) typical XPS spectra taken during depth profiling.

experiments. Quantities of Ga or As present in the bulk of the TiO₂ are so small as to be unobservable.

The TiO₂ layers are not perfectly uniform over the surface of the GaAs coupons; scanning electron micrographs reveal graininess of 1 μm or less at the surfaces of the films. Scanning Auger micrographs of low-humidity films show that the surface is uniformly covered by Ti and that Ga and As are not present. High-humidity films show Ti, Ga, and As present at the surface in the same regions to the resolution of the instrument (several micrometers). Thus the graininess of the micrographs is not due to separate islands of TiO₂ and native oxide unless the islands' diameters are smaller than several micrometers. The fast rise of the substrate XPS signals in the depth profiles indicates that, by the time the interface is reached, the etch procedure has smoothed out the film to a graininess of at most 75 Å. Nonuniform etching through the film (etch-pit formation), which causes patchy TiO₂ coverage near the interface, would reduce detection of interfacial native oxide from the usual XPS sensitivity of a fraction of a monolayer at the surface to a full monolayer or more at the interface, as discussed above. In the samples hydrolyzed in low humidity, then, there is at most one monolayer's worth of native oxide in the interfacial region defined by the depth resolution of the solution etch procedure (~50 Å).

IV. CONCLUSIONS

This study has demonstrated that a single-component, nonnative oxide coating can be formed at room temperature on GaAs without mixing of semiconductor and oxide, and without the formation of an intervening layer of native oxide (to within our detection limits of one monolayer). This result is significant, given the strong imputation to native oxide of electron traps that cause poor device performance. Single-component, nonnative oxide layers were not stable when stored in room air: native oxide was eventually formed at the air-nonnative oxide interface. Understanding the mechanism of chemical deposition of nonnative oxide films on GaAs and their subsequent reactions will enable an assessment of their value in the making of electronic devices.

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